

Conversions of Carboxylated Butadiene-styrene
Rubbers Under the Action of Gamma Radiation

81609

S/190/60/002/02/08/011
B004/B061

which is particularly heavy with a small radiation dose. The connection observed between the quantities of gel formed and carboxyl groups consumed indicates a complicated process of structure formation and destruction. The latter is seen in a decrease, especially rapid with small doses, of viscosity of the brine fraction. Intensive interlacing is caused by raising the methacrylic acid content. There is a linear relation between the number of carboxyl groups and the number of cross-links formed. The number of cross-links calculated from the data of the swelling agrees well with radiation doses of up to 20 Mr with the number calculated from the carboxyl groups consumed. There are 5 figures, 3 tables, and 6 references: 3 Soviet and 3 US.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii
(Moscow Institute of Fine Chemical Technology)

SUBMITTED: November 12, 1959

Card 2/2

AUTHORS:

Dogadkin, B. A., Shershnev, V. A., Dobromyslova, A. V.

TITLE:

Reversion Phenomena in the Vulcanization of Rubber With Tetramethylthiuramdisulfide

PERIODICAL:

Vysokomolekulyarnyye soyedineniya, 1960, Vol. 2, No. 4, pp. 514-517

TEXT: The authors investigated the vulcanization of natural rubber and CKM (SKI) isoprene rubber with tetramethylthiuramdisulfide at 143°C in the absence or in the presence of oxides of zinc, magnesium, or calcium in air and argon. Fig. 1 shows the change in the steric network in SKS rubber in air and argon as function of the duration of vulcanization; Fig. 2 shows the kinetics of sulfur accumulation, and Fig. 3 shows the change in the steric network in natural rubber as a function of the duration of vulcanization. Addition of phenyl-β-naphthylamine-1 as the cross-linking. A decrease of the density of the steric network was observed when natural rubber was vulcanized in air and argon. A reversion of vulcanization was observed when natural rubber was vulcanized in air and argon.

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Reversion Phenomena in the Vulcanization of
Rubber With Tetramethylthiuramdisulfide

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rubber or SKI-rubber was vulcanized with tetramethylthiuramdisulfide without metallic oxides or in the presence of magnesium- or calcium oxides (Table 1). In this case, the dimethyldithiocarbamic acid decomposes into hydrogen sulfide and dimethylamine. Although this decomposition was observed also in argon, no reversion occurred. In the presence of ZnO, reversion occurs neither in air nor in argon, because the dimethyldithiocarbamic acid is bound as zinc salt. Zinc increases also the stability of the vulcanizate to aging (Table 2). The authors explain the reversion of rubber vulcanization by destructive oxidation processes which are intensified by the decomposition products of dimethyldithiocarbamic acid, but are prevented by the binding of this acid with zinc. There are 3 figures, 2 tables, and 3 references: 1 Soviet.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov)

SUBMITTED: December 24, 1959

Card 2/2

TARASOVA, Z.N.; KAPLUNOV, M.Ya.; KOZLOV, V.T.; KLAUZEN, N.A.;
DOGADKIN, B.A.

Interaction of sulfur with natural rubber under the influence
of ionizing radiations. Vysokom. soed. 2 no.8:1201-1206
Ag '60. (MIRA 13:9)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti.
(Sulfur) (Rubber) (Gamma rays)

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D034/D002

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15.9120

AUTHOR:

Tarasova, Z.N., Dogadkin, B.A., Arkhangel'skaya, M.I.
Petrova S.B.

TITLE:

The Structure and Properties of Vulcanizates of
Carboxylated Rubber Produced by the Combined Action
of Metal Oxides and High Energy Radiation

PERIODICAL:

Kolloidnyy zhurnal, 1960, Vol XXII, Nr 2, pp 253-256
(USSR)

ABSTRACT:

On the basis of a number of investigations the authors
of the article discuss the effect of the structure
of vulcanizates of carboxylated rubber on their
strength properties. It could be established that
the rate constant of stress relaxation of these
vulcanizates at 150°C is about 50-100 fold that of
the vulcanizates with polysulfide bonds [Ref. 1].
Investigation of the change of osmotic and viscosi-

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D034/D002

The Structure and Properties of Vulcanizates of Carboxylated Rubber
Produced by the Combined Action of Metal Oxides and High Energy
Radiation

metric properties of rubber mixture and vulcanizate solutions prior to and after relaxation showed that the molecular weight does not considerably change. This in connection with the observed preservation of the number of cross links during relaxation suggests the conclusion that the weakening of the stress during the relaxation of carboxylated rubber vulcanizates with salt type cross bonds is due to the disintegration of the latter and the rising of new bonds as a result of exchange reactions. The low thermal stability of salt type bonds requires additional introduction of stable bonds into the vulcanization network. Good results were obtained with Co-60 treatment of carboxylated rubber preliminarily vulcanized

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The Structure and Properties of Vulcanizates of Carboxylated Rubber
Produced by the Combined Action of Metal Oxides and High Energy
Radiation

with metal oxides. The formation of a limited number
of cross bonds-C-C- (approximately 1 per 1000 mono-
mer units) permits preparing vulcanizates of high
thermal stability and strength. The strength of such
vulcanizates exceeds 400 kg/cm². There are 1 graph,
1 table and 4 references, 3 of which are Soviet
and 1 English.

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promysh-
lennosti, Moskva (Scientific Research Institute of
the Tire Industry, Moscow)

SUBMITTED: November 9, 1959

Card 3/3

S/069/60/022/005/006/011
B015/B064

AUTHORS: Lukomskaya, A. I. and Dogadkin, B. A.

TITLE: The Possibility of Studying the Structure of Vulcanizates Filled With Carbon Black by Measuring Their Dielectric Properties 21

PERIODICAL: Kolloidnyy zhurnal, 1960, Vol. 22, No. 5, pp. 576-586

TEXT: The present paper discusses published data on the change of the dielectric constant ϵ' and the loss factor ϵ'' of the frequency $f = \omega/2\pi$ or temperature T for vulcanizates filled with carbon black. In this connection it is found that the temperature - frequency functions may be used in the quantitative determined of vulcanizates. On the basis of the characteristics obtained, it is possible to form an idea of the structure of vulcanizates filled with carbon black. If the value of the dielectric constant at high frequency ϵ'_{∞} in dependence on the carbon-black content is known in a mixture p , the form factor Φ of the carbon-black particles can be determined: $\epsilon'_{\infty} = \epsilon'_r(1 + \Phi p)$ (4) (ϵ'_r = dielectric constant of the rubber phase). If Φ is independent of the carbon-black

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The Possibility of Studying the Structure of
Vulcanizates Filled With Carbon Black by
Measuring Their Dielectric Properties

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content, spherical carbon black particles are found at $\Phi = 1$, and extended ones at $F > 1$. A rise of Φ with the carbon-black content (Fig. 1) indicates the formation of a "structure" by the carbon-black particles (chain- or net structure). Since Φ changes in the same sense as the "structural" dielectric losses ϵ''_{str} , it is possible to determine the carbon-black structure from the experimental value ϵ'' . In the course of previous investigations (Refs. 13-16), the authors observed at high frequencies and low temperatures that ϵ''_{str} is in agreement with ϵ'' . At high temperatures and low frequencies (as well as in the case of direct current), it is possible to use conductivity for structure determination. Additional information on the structural changes of filled vulcanizates are obtained by direct measurement of ϵ' and ϵ'' during vulcanization. There are 6 figures and 27 references: 12 Soviet, 16 US, 8 British, 2 French, 4 German, and 1 Japanese.

ASSOCIATION:

Nauchno-issledovatel'skiy institut shinnoy promyshlennosti
(Scientific Research Institute of the Tire Industry)

SUBMITTED:

March 17, 1960

Card 2/2

PEASE I BOOK EXPILOTATION

International symposium on macromolecular chemistry. Moscow, 1960.

Mezhduarodnyy simpozium po makromolekulyarnoy khimii SSSR, Moskva, 14-18 iyunya 1960 g.: doklady i avtoreferaty. Sektziya III. (International Symposium on Macromolecular Chemistry Held in Moscow, June 14-18, 1960: Papers and Summaries) Section III. (Moscow, Izd-vo AN SSSR, 1960) 469 p. 55,000 copies printed.

Tech. Ed.: P. S. Kashina.

Sponsoring Agency: The International Union of Pure and Applied Chemistry. Commission on Macromolecular Chemistry.

PURPOSE: This book is intended for chemists interested in polymerization reactions and the synthesis of high molecular compounds.

COVERAGE: This is Section III of a multivolume work containing papers on macromolecular chemistry. The articles in general deal with the kinetics of polymerization reactions, the synthesis of special-purpose polymers, e.g., ion exchange resins, semiconductor materials, etc., ion ex-
alving polymerization reactions, properties and chemical interactions of high molecular materials, and the effects of various factors on polymerization and the degradation of high molecular compounds. No personalities are mentioned. References given follow the articles.

- Babek, T. I., and J. Kosmider (Poland). Chlorination of Phenol-Formaldehyde Resins 27
- Alexandru, L., M. Doris, and A. Cioconel (Rumania). Cyanoethyl and Aminoethyl Ethers of Polyvinyl Alcohol 34
- Yamborich, I. Ya., G. Ya. Gordin, L. I. Maslennikova, Ye. M. Gerasim, M. I. Krut'kova, and M. I. Kozlovskaya (USSR). Study of the Chemical Conversions of Polycarbonates 44
- Pyzdin, B. A., M. S. Fel'dbaum, and Z. M. Belyayeva (USSR). Characterization and Mechanism of the Activating Action of Double Systems of Vulcanization Accelerators 65
- Pinkus, I. M., A. P. Vorob'yeva, G. A. Shapokova, and M. P. Rodichayeva (USSR). Ethers of Sulfuric Acid and Polyvinyl Alcohol 73
- Volkóber, Z., T. Molly, and G. Zsurz (Hungary). The Inter-action of Aromatic Amines and Polyvinyl Chloride 79
- Gardner, M. A., B. E. Davison, B. A. Krenzel, J. M. Kue-nrich, L. S. Polak, A. V. Zorichyev, and R. M. Yozienko (USSR). The Production of Polymeric Materials Which Exhibit Semiconductor Properties 85
- Mikes, J. A., and L. I. Kovács (Hungary). Chemical Properties of Bipolar Ion-Exchange Resins 93
- Babek, T. I., and J. Kosmider (Poland). Effect of the Struc-ture of Organic Amino Compounds on the Properties of Anion Exchange Resins From Polystyrene 102
- Seliduck, K. M. (USSR). The Problem of the Effect of the Structure of Ions on Ion-Exchange Processes Between Ionites and Electrolyte Solutions 107
- Berlin, A. A., B. I. Magon'kii, and V. P. Patalin (USSR). Production and Properties of Some Aromatic Polymers 115
- Trostyanskaya, Ye. V., I. P. Losev, A. S. Terkina, S. B. Zakharova, O. Z. Metedova, and M. Hsien-jio (USSR). Chemical Conversions of Insoluble Copolymers of Styrene 124
- Lindeman, J. (Poland). Thermal Stability of Strongly Basic Anion Exchange Resins 146 40

PHASE I BOOK EXPLOITATION

International symposium on macromolecular chemistry
1960.

Mezhdunarodnyy simpozium po makromolekulyarnoy khimii SSSR, Moskva, 14-18 Iyunya 1960 g.; doklady i khlopyaty. Sektziya III. (International Symposium on Macromolecular Chemistry Held in Moscow, June 14-18, 1960; Papers and Summaries) Section III. [Moscow, Izd-vo AN SSSR, 1960] 469 p. 55,000 copies printed.

Tech. Ed.: P. S. Koshina.

Sponsoring Agency: The International Union of Pure and Applied Chemistry. Commission on Macromolecular Chemistry.

PURPOSE: This book is intended for chemists interested in polymerization reactions and the synthesis of high molecular weight compounds.

COVERAGES: This is Section III of a multivolume work containing papers on macromolecular chemistry. The articles in general deal with the kinetics of polymerization reactions, the synthesis of special-purpose polymers, e.g., ion exchange resins, semiconductor materials, etc., ion-exchanging polymerization reactions, properties, methods of characterization, and interactions of high molecular materials, and chemical various factors on polymerization materials, and the effects of high molecular compounds. The degradation and the degradation of references given follow the articles. No personalities are mentioned.

Ushakov, Kh. U., U. N. Muzayev, and R. S. Tilyayev (USSR).
The Radiation Method of Copolymerizing Acrylonitrile with
Polystyrene and Perchloroethylene. *Chem. Abstr.* 1966, 62, 12510d.

Radikov, S. B.,
Ozobkova (USSR),
Chain Polyamides
G. N. Chelnokova,
Oxyethylation of Carbochain and Hetero-

Santo, I., and K. Gal (Hungary). Grafting Methyl Methacrylate onto Films of Polyvinyl Alcohol. <i>Alcohol Chemistry and Technology of Carbochain and Hetero-chain Polyamides</i>	184
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Lazar, M., R. Radov and Tu. Pavlineta (Czechoslovakia),
Grafting Methyl Methacrylate Onto Polyethylene
with α -Vinyl Alcohol Under the Action of X-Rays 207

Tutornik, V. A.; Zolotarev, and V. M. Bystryak (USSR).
Interaction of Carbonyl-Containing Butadiene-Styrene
Rubbers With Polyamides and E-Caprolactam

224

Source of Free Radicals on Crosslinking in Polyethylene. The Role of the Oxidation Products of Carboxylic Acids (A. A. Boradkin, V. I. Kuznetsov, and N. P. Gerasimov). *Dokl. Akad. Nauk SSSR*, 1978, No. 6, p. 1095.

styrene rubbers and their mixtures with ϵ -caprolactam under the action of gamma radiation).

egorov, Z. A., V. A. Derzitskaya, Sun T'ung, Chang Wei-
ang, and L. S. Gal'brayn (USSR): Synthesis of New
Lactam Derivatives and Other Polymers. *Chem. Abstr.* 1963, 57, 12482c.

Armenenko, I. M., and P. M. Kaputsky (USSR). Initiation of the Controlled Synthesis of Modified Celluloses of Nitrogen Derivatives and Other Polysaccharides

NOV, V. I., M. Ye. Lemshina, V. S. Ivleva (USSR).
Additional Transformations in Chains of Cellulose.
Ilin, A. A.

ilin, A. A., Ye. L. Pentskaya, and O. I. Volkova (USSR).
 Functional Chemical Transformations and Block Copolymeriza-
 tion During the Freezing of Starch Solutions.

NOV. 20, 1966 B. I. AYKHODZHAYEV, and N. AIZYER (USSR).
Classification of the Properties of Cellulose by Grafting
Polymers onto Starch Solutions 334

... by Grafting

S/069/60/022/006/001/008
B013/B066

AUTHORS:

Dogadkin, B. A., Skorodumova, Z. V., and Fel'dshteyn, M. S.

TITLE:

Effect of the Chemical Nature of the Surface of Carbon Black on Its Interaction With Rubber and Sulfur, and on the Vulcanization Kinetics

PERIODICAL:

Kolloidnyy zhurnal, 1960, Vol. 22, No. 6, pp. 663-670

TEXT: The purpose of the present paper was to study the interaction of carbon black with rubber and the dependence of this reaction on the nature of the carbon-black surface. The interaction in the systems rubber - carbon black and rubber - carbon black - sulfur was studied in butadiene-styrene rubber CKC-30A(SKS-30A). The vulcanization temperature was 143°C. The sorption of rubber from n-heptane solutions (Fig. 1) indicated that the commercial blacks drop in the following order according to the quantity of rubber sorbed per unit surface: Lampblack > thermal black > furnace black > channel black. The type "Feelblack O" corresponds to channel black. The rubber quantity sorbed per surface unit

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Effect of the Chemical Nature of the Surface
of Carbon Black on Its Interaction With Rubber
and Sulfur, and on the Vulcanization Kinetics

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is the higher, the less oxygen-containing functional groups occur on the black surface. The interaction of rubber with carbon black permitted the establishment of a similar relationship at vulcanization temperature. It was shown that the sulfur chemically bound on the black surface forms additional active centers, and participates in the formation of cross links. Since the opinions on the character of the interaction of carbon black with rubber diverge, this problem requires further thorough investigation. The effect of the oxidation of carbon black on the vulcanization kinetics was tested on the type "Feelblack 0" which is used to a considerably extent in the tire industry. It was oxidized for 1.5 hours at 400°C in the air. The oxygen content in the carbon black increased and the low pH was indicative of an increased content of carboxyl and phenol groups. It was found that the increased number of oxygen-containing functional groups on the surface of carbon black reduce the vulcanization rate, and the moduli, the content of bound sulfur, and increase the maximum of swelling. This effect of oxygen-containing functional groups was also confirmed by the data obtained for sulfur by heating the system rubber - carbon black - sulfur with contents of lampblack, channel black,

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Effect of the Chemical Nature of the Surface
of Carbon Black on Its Interaction With Rubber
and Sulfur, and on the Vulcanization Kinetics

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"Feelblack O", and furnace black (Fig. 6). Samples of channel black
which had been subjected to heat treatment were made available by
B. V. Lukin and K. A. Pechkovskaya. There are 6 figures, 4 tables, and
12 references: 8 Soviet, 7 US, 1 British, and 2 Australian.

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promyshlennosti,
Moskva (Scientific Research Institute of the Tire Industry,
Moscow)

SUBMITTED: June 6, 1960

Card 3/3

TARASOVA, Z.N., DOGADKIN, B.A., ARKHANGEL'SKAYA, M.I., PETROVA, S.B.

Structure and properties of vulcanizates produced from carboxylated polymers obtained through the combined action of metal oxides and high energy radiation. Koll. zhur. 22 no.2:253-256
Mr-Apr '60. (MIRA 13:8)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti, Moskva.

(Rubber—Research) (Radiation)

FEL'DSHTEYN, M.S.; ORLOVSKIY, P.N.; DOGADKIN, B.A.

Effect of accelerators as determined by the temperature of vul-
canization. Kauch.i rez. 19 no.12:27-31 D '60. (MIRA 13:12)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti.
(Vulcanization)

DOGADKIN, B.A.; SKORODUMOVA, Z.V.; FEL'DSHTEYN, M.S.

Effect of the chemical nature of a carbon-black surface on its interaction with rubber and sulfur and on the vulcanization kinetics. Koll. zhur. 22 no. 6:663-670 N-D '60. (MIRA 13:12)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti, Moskva.

(Carbon black) (Vulcanization)

DOGADKIN, B.A.

PHASE I BOOK EXPLOITATION

SOV/5486

137

Vsesoyuznoye soveshchaniye po vnedreniyu radioaktivnykh izotopov i yadernykh izlucheniyy v narodnoye khozyaystvo SSSR. Riga, 1960.

Radioaktivnyye izotopy i yadernyye izlucheniya v narodnom khozyaystve SSSR; trudy soveshchaniya v 4 tomakh. t. 1: Obshchiye voprosy primeneniya izotopov, pribory i istochniki radioaktivnykh izlucheniyy, radiatsionnaya khimiya, khimicheskaya i neftepererabatyvayushchaya promyshlennost' (Radioactive Isotopes and Nuclear Radiations in the National Economy of the USSR; Transactions of the Symposium in 4 Volumes. v. 1: General Problems in the Utilization of Isotopes; Instruments With Sources of Radioactive Radiation; Radiation Chemistry; the Chemical and Petroleum Refining Industry) Moscow, Gostoptekhizdat, 1961. 340 p. 4,140 copies printed.

Sponsoring Agency: Gosudarstvennyy nauchno-tekhnicheskii komitet Soveta Ministrov SSSR, and Gosudarstvennyy komitet Soveta Ministrov SSSR po ispol'zovaniyu atomnoy energii.

Ed. (Title page): N.A. Petrov, L.I. Petrenko and P.S. Savitskiy; Eds. of this Vol.: L.I. Petrenko, P.S. Savitskiy, V.I. Sinitsin, Ya. M. Kolotyarkin, N.P. Syrkina and R.F. Romm; Executive Eds.: Ye. S. Levina and B. F. Titkaya; Tech. Ed.: E.A. Mukhina.

Card 1/10

Radioactive Isotopes (Cont.)

807/5486

137.

PURPOSE: The book is intended for technical personnel concerned with problems of application of radioactive isotopes and nuclear radiation in all branches of the Soviet economy.

COVERAGE: An All-Union Conference on problems in the introduction of radioactive isotopes and nuclear radiation into the national economy of the Soviet Union took place in Riga on 12-16 April 1960. The Conference was sponsored by: the Gosudarstvennyy nauchno-tekhnicheskii komitet Soveta Ministrov SSSR (State Scientific and Technical Committee of the Council of Ministers, USSR); Glavnoye upravleniye po ispol'zovaniyu atomnoy energii pri Sovete Ministrov SSSR (Main Administration for the Utilization of Atomic Energy of the Council of Ministers, USSR); Academy of Sciences, USSR; Gosplan USSR; Gosudarstvennyy komitet Soveta Ministrov SSSR po avtomatizatsii i mashinostroyeniyu (State Committee of the Council of Ministers, USSR, for Automation and Machine Building) and the Council of Ministers of the Latvian SSR. The transactions of this Conference are published in four volumes. Volume I contains articles on the following subjects: the general problems of the Conference topics; the state and prospects of development of radiation chemistry; and results and prospects of applying radioactive isotopes and nuclear radiation in the petroleum refining and chemical industries. Problems of designing and manufacturing instruments which contain sources of radioactive radiation and are used for checking and automation of technological processes are examined, along with problems of accident prevention in their use. No personalities are mentioned. References accompany some of the articles.

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Radioactive Isotopes

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Korablev, L.N. Specifications of Tubes and Cold Cathodes

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RADIATION CHEMISTRY

Breger, A. Kh. Sources of γ -Radiation for Radiation-Chemical Apparatus

169

Syrkus, N.P., A.Kh. Breger, and B.I. Vaynshteyn. Basic Technological Characteristics of a Potential Apparatus for Carrying Out Radiation Polymerization of Ethylene on an Industrial Scale

176

Dogadkin, B.A., Z.N. Tarasova, M. Ya. Kaplunov, A. Kh. Breger, L.M. Kapersha, B.I. Vaynshteyn, Ya. M. Vizel', and V.L. Karpov. Intensification of the Process of Radiation Vulcanization and the Technical Principles of an Experimental Installation for the Radiation Vulcanization of Tires

184

Dzhagatspanyan, R.V., V.I. Zetkin, G.V. Motsarev, and M.T. Filippov. Chlorination of Silicon-Containing Monomers and Polymers Under the Action of γ -Radiation

197

Card 7/12

11.2211
15.9300
AUTHORS:

34896
S/081/62/000/003/085/090
B 162/B101
Dogadkin, B. A., Tarasova, Z. N., Kaplunov, M. Ya., Brager,
A. Kh., Kopersha, L. M., Vaynshteyn, B. I., Vize'l', Ya. M.,
Karpov, V. L.

TITLE:

Intensification of the process of radiation vulcanization
and technical principles of an experimental installation for
radiation vulcanisation of tyres

PERIODICAL:

Referativnyy zhurnal. Khimiya, no. 3, 1962, 595 - 596,
abstract 3P275 (Sb. "Radioakt. izotopy i yadern.izlucheniya
v nar. kh-ve SSSR, v. I", M., Gostoptekhzdat, 1961, 184-196)

TEXT: An investigation was made into the effect of medium (air and vacuum),
temperature (from -196 to 100°C), sensitizers and inhibitors on radiation
vulcanization under the action of Co^{60} γ - radiation of butadiene,
butadiene-styrene and natural rubber. The degree of cross-linking in air
is higher than in vacuum. In the presence of 2 % phenyl - β - naphthyl-
the radiation-chemical yield of cross-links per 100 ev of absorbed

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B162/B101

Intensification of the process ...

energy drops by half for butadiene rubber in vacuum. The decrease in non-saturation is only partially explained by cross-linking and oxidation, and in the main this phenomenon is probably connected with the formation of intra-molecular rings. The cross-linking at different temperatures depends to a large extent on the structure of the rubber. Aliphatic polyhalides reduce the required radiation dose by half (to 25 Mr) and ensure the production of rubbers with a static strength equal to the strength of the best sulphur vulcanized rubbers. Vulcanization of rubbers containing carboxyl by the combined action of metal oxides and nuclear radiation (dose 10 Mr) gives vulcanized rubbers with high thermal stability and high strength properties. An investigation was made into the kinetics of the addition of styrene and 2,5 -dichlorostyrene to natural rubber and butadiene-styrene rubber and to mixtures of these with channel black with irradiation in Ar. An acceleration of vulcanization was observed in the presence of these monomers and vulcanized rubbers were obtained which possessed high thermomechanical stability and strength. The technical principles of a technological process for an experimental installation for radiation vulcanization of tyres are examined. Different types of γ -radiation sources were compared: radiation In-Ga loop of a nuclear reactor,

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Intensification of the process ...

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spent-fuel assemblies, Co⁶⁰ and different types of irradiators. A scheme is proposed for a technological process for an experimental installation with spent-fuel assemblies. [Abstracter's note: Complete translation]

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Card 3/3

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S/138/61/000/009/004/011
A051/A129

15.9130

AUTHORS: Tarasova, Z. N., Eytingon, I. I., Senatorskaya, L. G., Fedorova, T. V.,
Dogadkin, B. A.

TITLE: Application of phenothiazine (thiodiphenylamine) as an antifatigue
agent of NR, CKM (SKI) and CKC-30AM (SKS-30AM) vulcanizates

PERIODICAL: Kauchuk i rezina, no. 9, 1961, 15 - 18

TEXT: A study was carried out to determine the action of phenothiazine during the vulcanization and fatigue of NR, SKI and SKS-30AM rubbers. It was established that phenothiazine has no significant effect on the kinetics of vulcanization and on the standard physico-mechanical properties of the vulcanizates. It increases the durability of the vulcanizates from the given rubbers during the process of repeated deformations under various conditions of fatigue. Phenothiazine or the products of its transformation combine with the vulcanizate under the effect of thermo-oxidizing action and repeated deformations. No combining of phenothiazine was noted during the process of thermal action alone. Phenothiazine in conjunction with certain oxidation inhibitors has more than just an additive action (mutually-intensifying action). A study of the exchange ability of the

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A051/A129

Application of phenothiazine...

vulcanizates with elemental sulfur showed that phenothiazine does not affect the nature of the vulcanizing structures, and during vulcanization at 143°C causes noticeable changes in the type of the sulfur bonds at temperatures of 173°C. A further study of its ability to react in isotope exchange with elemental sulfur showed that under vulcanization at 173°C there is no noticeable sulfur exchange in phenothiazine. Data of Table 1 reveal that phenothiazine reduces the rate of chemical relaxation by 3 to 7 times in NR vulcanizates and by a factor of two in vulcanizates of SKI, and by 2 - 3 times in SKS-30A vulcanizates. It has a more effective action in rubbers produced at elevated vulcanizing temperatures than other known anti-fatigue agents, such as N-phenyl-N'-cyclohexyl-n-phenylenediamine (4010). Phenothiazine increases the durability of the vulcanizates during the process of repeated deformations in symmetrical sign-changing loading and in repeated bending. It reacts with the products of oxidation, stabilizing the latter and thus preventing the further development of the thermo-oxidizing destruction. The application of a system of inhibitors having a combined intensifying action shows promise in extending the service life of rubbers and stabilizing them. There are 2 tables, 1 set of graphs and 9 references: 6 Soviet-bloc and 3 non-Soviet-bloc. The references to the English-language publications read as follows:

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A051/A129

Application of phenothiazine...

Murphy, Ravner, Smith, Ind. Eng. Chem., 42, no. 2, 2479 (1950); A. Tobolsky, J. Appl. Phys., 27, no. 7, 673 (1956).

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promyshlennosti (Scientific Research Institute of the Tire Industry)

Table 1. Effect of the type of the anti-fatigue agent introduced into the mixture on the rate of chemical relaxation of tension and the durability of the vulcanizates during the fatigue process (dosage of anti-fatigue agent 1.0 w.p. to 100 w.p. of rubber)

Type of rubber	Type of anti-fatigue agent	Vulcanization conditions		Rate of relaxation constant at 130°C, min. ⁻¹ · 10 ⁻³		Durability in deformations, 1,000 cycl.	
		temp., °C	duration, min.	in air	in non-oxygen conditions	symmetr. loading at 100°C	repeated bending pinning at 20°C
NR	without anti-fatigue agent	143	20	38.0	1.11	1,934	-
	phenothiazine . . .	143	20	11.5	1.07	3,217	-

Card 3/4

S/190/61/003/004/001/014
B101/B207

AUTHORS: Dogadkin, B. A., Dobromyslova, A. V., Belyatskaya, O. N.,
Gyul'-Nazarova, T. A.

TITLE: Study of the early vulcanization (scorching) of rubber mix-
tures. 1. Structural changes of non-filled and filled mix-
tures when heated

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 3, no. 4, 1961,
497-504

TEXT: The present study deals with the structural changes occurring in the scorching of rubber mixtures as well as with the effect of various factors upon this process. The investigation was conducted by means of a plastometer of the NII ShP (Scientific Research Institute of the Tire Industry) at 120°C. The mixtures were heated in the plastometer for seven minutes and then, at constant pressure, pressed through a capillary; every two minutes, the quantity leaving the capillary was weighed. The moment at which no more mixture left the capillary, was defined as scorching point. Preliminary tests proved that the data obtained by means of the plastometer are in good

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B101/B207

agreement with those obtained by the BP-1 (VR-1) viscosimeter. Moreover, the kinetics of sulfur addition and the change of the solubility in benzene were tested. A) Non-filled mixtures. The experiments were made with a mixture of (in weight %) 100 CMC-30A (SKS-30A) rubber, 3 sulfur, 1.2 N,N-diethylbenzothiazyl sulfenamide, 1.2 dibenzothiazyl disulfide, 2 zinc oxide, 2.0 stearic acid. Fig. 1 shows the results obtained. The curve of S addition does not go through the origin of coordinates, since the initial rubber contains already 0.2% S. In the scorching point, the S addition amounts to about 0.5%. B) Filled mixtures. Carbon black served as filler. The mixture consisted of (in weight %) 100 SKS-30A rubber, 3.0 sulfur, 1.2 sulfenamide ET (BT), 1.2 altax, 5.0 ZnO, 1.0 colophonium, 3.0 rubrax, 1.0 stearic acid, 5.0 polydienes, 40 spray burner black, 15.0 carbon black. The results are listed in Fig. 2. In the presence of highly surface-active carbon black, the scorching point occurred already after the addition of 0.25-0.30% sulfur, while in the presence of coarse-disperse carbon black, 0.4-0.5% S is added. C) The authors studied the effect exerted by various types of carbon black the properties of which are listed:

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B101/B207Table 1.

Type of carbon black	pH of the carbon black suspension in water-alcohol mixture	specific surface m ² /g	scorching time min
Carbon black	3.2 - 3.3	90	94
Carbon black, reduced	8.4 - 8.6	-	62
Chimney soot	8.0 - 8.2	30	62
Chimney soot oxidized	6.2 - 6.4	-	98
Spray burner black	7.4 - 7.6	25	70
Thermal carbon black	7.4 - 7.6	15	76

Fig. 4 shows the effect of the pH of carbon black upon the sulfur addition. The effect of the degree of dispersion of carbon black manifested itself by the fact that carbon black, already when masticated with rubber, forms rubber - black gel (approximately 42%), while in the case of coarse-disperse

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chimney soot this effect was not observed. Heating of carbon black in N_2 to eliminate the oxygen-containing groups, had no effect upon this phenomenon, led, however, due to the pH increase, to a quicker sulfur addition. The network formation with fine-disperse carbon black was also observed in mixtures with natural rubber. Thus, scorching is caused by interaction of rubber with sulfur and other vulcanizing substances. The only means of a successful elimination is an inhibition of the mentioned processes. V. A. Zhukova participated in the experiments. There are 6 figures, 2 tables, and 14 references: 2 Soviet-bloc and 12 non-Soviet-bloc. The 2 references to English language publications read as follows: M. L. Studebaker, L. G. Nabors, Rub. Age 80, 5, 837, 1957; W. H. Watson, Industr. and Engng. Chem. 47, 1281, 1955.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov). NII shinnoy
promyshlennosti (Scientific Research Institute of Tire Industry)

SUBMITTED: June 10, 1960

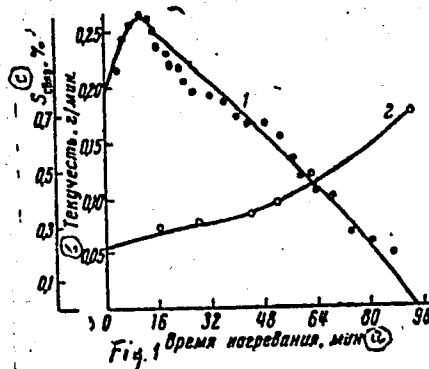
Card 4/7

S/190/61/003/004/001/014
B101/B207

Study of ...

Fig. 1: Change of the flow and the amount of bound sulfur when heating non-filled butadiene-styrene rubber to 120°C.

Legend: 1) flow; bound sulfur; a) time of heating; min; b) flow; c) bound sulfur, %.



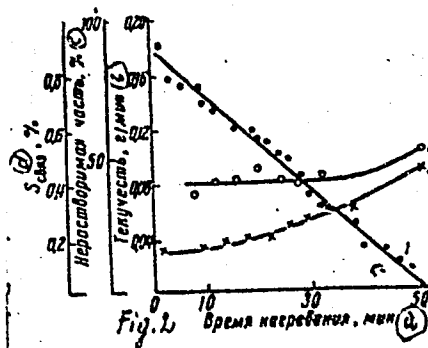
Card 5/7

Study of ...

Fig. 2: Change of the flow, solubility and amount of bound sulfur when heating filled rubber to 120 °C.

Legend: flow; 2) solubility; 3) bound sulfur; a) time of heating, min; b) insoluble portion; c) bound sulfur.

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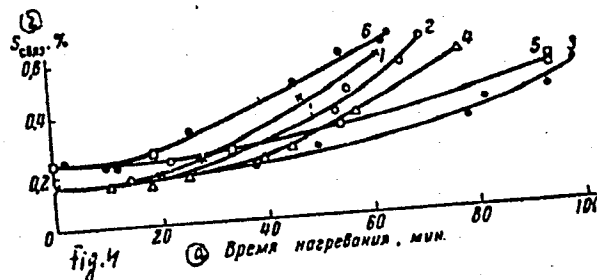


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S/190/61/003/004/001/014
B101/B207

Study of ...

Fig. 4: Kinetics of the sulfur addition to rubber mixtures.
Legend: 1) chimney soot (pH = 8);
2) spray burner black (pH = 7.4);
3) chimney soot oxidized (pH = 6.2);
4) thermal carbon black (pH = 7.4);
5) carbon black (pH = 3.2);
6) carbon black reduced (pH = 8.4);
a) time of heating; b) bound sulfur.



Card 7/7

DOGADKIN, B.A.; PAVLOV, N.N.; Primala uchastiye: RUMYANTSEVA, F.A.

Spectral study of the vulcanization of rubber. Vysokom.sped. 3
no.4:613-617 Ap '61. (MIRA 14:4)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V.
Lomonosova.
(Vulcanization--Spectra)

22566

S/190/61/003/005/009/014
B110/B220

15.9000

1436, 2209

AUTHORS:

Dogadkin, B. A., Tutorskiy, I. A., Tugov, I. I.,
Al'tzitzer, V. S., Krokhina, L. S., Shershnev, V. A.

TITLE:

The chemical modification of vulcanizates. I. The reaction
of vulcanizates with styrene, methyl methacrylate, and
isoprene

PERIODICAL:

Vysokomolekulyarnyye soyedineniya, v. 3, no. 5, 1961,
729-733

TEXT: The chemical modification of vulcanizates is completely new and
hardly mentioned in literature. The purpose of the present paper was to
study the chemical modification process caused by copolymerization of the
vulcanizates with the monomer. Natural rubber (I) or a mixture of natural
rubber and butadiene styrene rubber CKC-30 (SKS-30) (II) were disintegrated
to particles of about 1 mm, scrubbed in the Soxhlet with acetone, and
filled into a weighed ampulla. The monomer (purified styrene, methyl
methacrylate, or isoprene) was added in quantities assuring the uniform
swelling of the vulcanizate. Then the ampulla was sealed and heated in

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The chemical...

an oil thermostat. Conversion of monomer and yield in graft polymer were determined by weight. The product of copolymerization was extracted with the hot solvent of the formed homopolymer: methyl ethyl ketone for polystyrene, acetone for polymethyl methacrylate, benzene for polyisoprene. In order to initiate the copolymerization process the vulcanizates were ozonized first of all in a suspension of CCl_4 to introduce functional (probably peroxide) groups. One has made use of the ozonizer developed by the Kafedra gazovoy elektrokhimii MGU im. Lomonosova (Department for Gas Electrochemistry of the Moscow State University imeni Lomonosov). The experimental temperatures were: 60, 100, 110, 150, and 180°C. The curves of kinetic copolymerization of non-ozonized I and II are represented in Figs. 2a and 5. In case the vulcanizate had been ozonized previously, a large fraction of the isoprene added polymerized already at 60°C. A considerable part of the polymerized isoprene forms with the vulcanizate a graft polymer (Fig. 6). Also for the copolymerization of methyl methacrylate with vulcanizate, its previous ozonizing raises the reaction rate and yield in graft polymer (Fig. 7). The active centers of the rubber existing in the vulcanizate (double bonds and α -methylene groups)

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are able to act as branching points in the chain of the trimeric polymer and, thus, form the graft polymer. Moreover, the initial polymerization may be effected by oxygen-containing groups existing on the surface of the crushed vulcanizate. The surface increase effected by adsorption of monomers on the crushed polymerizate also accelerates the reaction. When polymerizing the non-ozonized vulcanizates with styrene at 150-180°C, the polymerization reaches its maximum already after the first 2 to 3 hr and then remains constant, since the thermopolymerization of styrene is practically completed. With a decrease in temperature of polymerization the yield in copolymers increases as compared to the total monomer polymerized. Yu. M. Yemel'yanov assisted in the experiments. There are 7 figures and 8 references: 3 Soviet-bloc and 5 non-Soviet-bloc. The two references to English-language publications read as follows:
Ref. 1: R. I. Ceresa, W. F. Watson, Trans. and Proceed 35, 19, 1959.
Ref. 4: I. Green, E. F. Sverdrup, Industr. and Engng. Chem. 48, 2138, 1956. X

Card 3/8

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S/190/61/003/005/009/014
B110/B220

The chemical...

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. Lomonosova (Moscow Institute of Fine Chemical Technology imeni Lomonosov) Vsesoyuznyy nauchno-issledovatel'skiy institut plenochnykh materialov i iskusstvennoy kozhi (All-Union Scientific Research Institute of Film Materials and Artificial Leather)

SUBMITTED: July 25, 1960

Fig. 2: kinetics of copolymerization: Legend: a) Vulcanizate of natural rubber with styrene; b) vulcanizate of natural + SKC-30 rubber with styrene. Full-line curves = styrene conversion; broken-line curves = yield in graft polystyrene. Temperature of polymerization: 1) = 110°C; 2) = 150°C; 3) = 180°C. c) time of polymerization, hr.

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S/190/61/003/010/017/019
B124/B110

// 22 //

AUTHORS: Dogadkin, B. A., Dobromyslova, A. V., Belyatskaya, O. N.

TITLE: Study of premature vulcanization (scorching) of rubber mixtures. II. Effect of inhibitors (antiscorchers) on the kinetics of sulfur addition to rubber

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 3, no. 10, 1961, 1572-1579

TEXT: The authors studied the effect of the best-known antiscorching agents (benzoic and phthalic acids, phthalic anhydride, and N-nitrosodiphenyl amine (NDPA)) on the vulcanization rate of rubber mixtures with sulfur. The rubber mixtures, filled later with carbon black, consisted of 100 parts by weight of styrene butadiene rubber CKC-30A (SKS-30A), 3.0 sulfur, 1.2 N,N-diethyl-amino-benzothiazole sulfenamide, 1.2 di-benzothiazyl disulfide, 5.0 ZnO, 2.0 stearic acid, and 1 part by weight of the antiscorchers mentioned. Table 1 shows the effect of the inhibitors examined on the time of scorching and the addition rate of sulfur to rubber. All antiscorchers prolong the time of liquid state of the mixture.

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Study of premature vulcanization ...

The inhibiting effect of NDPA is not affected by the presence or type of the accelerator (N-cyclohexyl benzothiazyl sulfenamide (CHBTS), diphenyl guanidine, 2-mercapto benzothiazole, tetramethyl-thiuram disulfide, N-oxy-diethylene-benzothiazyl sulfenamide (ODBTS), or 2,4-dinitro-phenyl ether of 2-mercapto benzothiazole (MBTDNP)). Mixtures of oil containing styrene butadiene rubber CKC-30AM-15 (SKS-30AM-15) were tested. The induction period in the addition of sulfur in mixtures containing CHBTS is increased in the presence of NDPA; the addition proceeds very slowly in the presence of ODBTS and MBTDNP at 120°C, and is still very small after 2 hr at 130°C. In mixtures on the basis of extracted butadiene styrene, vulcanization is not accelerated at 100°C in the presence of NDPA, even after 8 hr. A first-order equation holds for the interaction of sulfur with rubber; there is a linear dependence of the logarithm of the free-sulfur concentration on the time of heating. The constants of the reaction rate were calculated from a first-order equation (Table 2). The rate of addition of CHBTS and Thiuram to rubber is increased by NDPA; the shape of the kinetic curve for CHBTS addition is not changed by NDPA; only the amount of Thiuram sulfur bound to rubber rises. In conclusion, it may be stated that NDPA inhibits the interaction of sulfur with rubber. The mechanism of action of the other inhibitors examined is different, and depends on the type of

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Study of premature vulcanization ...

inhibitor used and the components of the rubber mixture. Thermal decomposition ($> 100^{\circ}\text{C}$) of NDPA produces NO which reacts in vulcanization with sulfur-containing radicals in the mixture, and inhibits rubber vulcanization. The second radical (diphenyl nitrogen) formed in thermal decomposition of NDPA, however, as a weak accelerating effect on the addition by accepting hydrogen. The dropping inhibitory effect of NDPA with increasing temperature dissociate to active particles with reduced number of sulfur atoms. The inhibition of crosslinking by NDPA is possibly not only due to the dropping addition rate of sulfur but also to the destructive effect of NDPA on the rubber. A. Ye. Grinberg et al. (Ref. 3: *Kauchuk i rezina*, 1959, no. 1, 22) and V. I. Gol'danskiy (Ref. 6 *Uspekhi khimii*, 15, 63, 1946) are mentioned. There are 8 figures, 2 tables, and 7 references: 5 Soviet and 2 non-Soviet. The two references to English-language publications read as follows: D. Craig, *Rubber Chem. and Technol.* 30, 1291, 1957; L. A. K. Staveley, C. N. Hinshelwood, *Trans. Faraday Soc.* 35, 845, 1939.

Card 3/6

28187

Study of premature vulcanization ...

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B124/B110

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov)

SUBMITTED: December 16, 1960

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158060

29744
S/190/61/003/011/016/016
B110/B147

AUTHORS: Dogadkin, B. A., Dontsov, A. A.

TITLE: Reaction of polyethylene with sulfur

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 3, no. 11, 1961,
1746-1754

TEXT: The results of the reaction of polyethylene (PE) with sulfur are given in the present paper. Nonstabilized PE ($[\eta]$ (tetralin, 135°C) = 1.94; $d_{25} = 0.915$ g/cm³) was mixed with sulfur, repeatedly recrystallized from benzene, in the laboratory mixer for rubber at 110°C-120°C. 2 g of mixture was heated in an ampul with Ar atmosphere in the oil bath. Content of H₂S, free and bound S; amount of gel; unsaturation, maximum of swelling, and the weight increase during swelling were determined in the reaction product. Heating of PE with S at 200°C-250°C produces binding of S with hydrocarbon, separation of H₂S, increase of the double bonds, formation and gradual increase of chemical cross links between the

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S/190/61/003/011/016/016
B110/B147

Reaction of polyethylene with sulfur

PE molecule chains. This causes gel formation. Cyclic structures are formed and small amounts of decomposition products of low molecular weight are separated. In the larger, rectilinear part of the kinetic curves for S addition, the absolute rate of reaction increases linearly with the initial S content. The relative rate $S_{\text{bound}}/S_{\text{total}}$ is inversely proportional. With an S content $> 6\%$ in the polymer, it becomes constant at 230°C , independent of the initial S content. The maximum amount of S ($31-37\%$) of the initial content is independent of the reaction temperature. The temperature coefficient of S addition is 2.44 , the activation energy 44.4 kcal/mole , and the pre-exponential factor in the Arrhenius equation $1.58 \cdot 10^{15} \text{ sec}^{-1}$, which corresponds to substitution reactions. The kinetic curves for the H_2S separation show a salient point which appears the quicker, the smaller the amount of bound S and the higher the reaction temperature. At a content of bound S $\leq 1.2\%$, no salient point occurs. Before the salient point the amount of separated H_2S is about equivalent to that of bound S. The ratio $\text{H}_2\text{S}/S_{\text{bound}}$ increases to ~ 2 towards the end of the reaction. H_2S develops, therefore, (1) because of

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S/190/61/003/011/016/016
B110/B147

Reaction of polyethylene with sulfur

primary reactions of S and PE, and (2) on account of secondary reactions of the bound S. (2) occurs at sufficient concentration of the S-containing groups and higher temperatures. To verify this, a mixture with 7.74 % S was heated at 230°C for 2-3 hr. After removal of free S by means of acetone, further heating was conducted at 230°C in Ar atmosphere for 2-3 hr. H₂S separation occurred with an increase of cross links in the insoluble fraction. The maximum amount of separated H₂S amounts to ~2/3

of the initial S. Accumulation of the double bonds occurs at a constant rate, depending on temperature and S content in the mixture. For

PE+7.74% S, $k_{230} = 4.64 \cdot 10^{-3} \text{ min}^{-1}$ (sulfur addition: $k_{230} = 4.16 \cdot 10^{-3}$).

A linear dependence exists between unsaturation increase and S addition. The total number of double bonds at the end of the process is 5-7.5 % of the equivalent of separated H₂S. Gel formation increases with increasing

reaction temperature and increasing initial S content, the maximum content of insoluble fraction, however, remains almost constant. It is reached at 0.6-0.7% of bound S. With increasing heating and S addition, the maximum of gel swelling in boiling toluene drops until termination of the

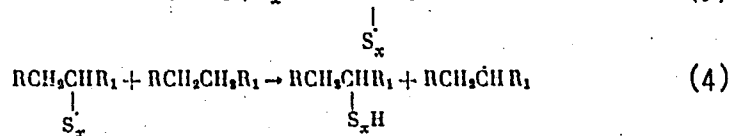
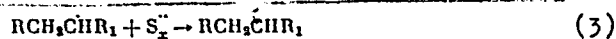
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B110/B147

Reaction of polyethylene with sulfur

S addition. A linear dependence exists between the maximum of swelling and the amount of bound S. The cross links probably develop owing to secondary regroupings of S bonds. The final concentration of cross links is $5.9-9.4 \cdot 10^{18}/\text{cm}^3$ (50-80 S atoms per cross link). Intramolecular S-containing cycles probably develop, or destruction processes occur. The structural changes are indicative of thermal destruction according to:

$S_8 \rightleftharpoons S_8^{\bullet\bullet} \rightarrow S_x^{\bullet\bullet} + S_y^{\bullet\bullet}$. The bi-radicals separate H from methine- or methylene groups: $RCH_2CH_2R_1 + S_x^{\bullet\bullet} \rightarrow RCH_2C \cdot HR_1 + HS_x^{\bullet}$. The following takes place:

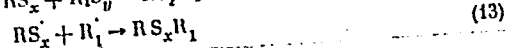
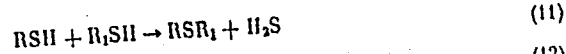


The structuration reaction occurs according to

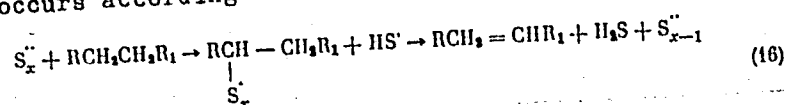
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Reaction of polyethylene with sulfur

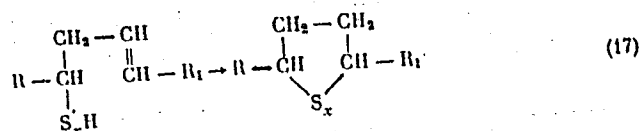
29744
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Double binding occurs according to



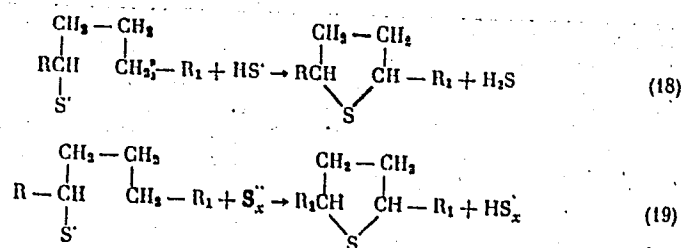
Ring formation proceeds according to



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Reaction of polyethylene with sulfur

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B110/B147



The authors thank A. V. Nikanorenkova for assistance with experiments. There are 7 figures and 11 references: 1 Soviet and 10 non-Soviet. The three most recent references to English-language publications read as follows: G. Gee, Trans. Faraday Soc., 48, 515, 1952; F. Faibrother, G. Gee, T. Merril, J. Polymer Sci., 16, 459, 1955; D. M. Gardner, G. K. Fraenkel, J. Amer. Chem. Soc., 78, 3279, 1956.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

SUBMITTED: January 4, 1961
Card 6/6

TARASOVA, Z. N.; KAPLUNOV, M. Ia.; KOZLOV, T. V.; KLAUZEN, N. A.; DOGADKIN, B.A.

Interaction of sulphur and natural rubber under ionizing radiation.
Chem prum 11 no.11:601-604 N '61.

1. Vyskumny ustav prumyslu pneumatik, Moskva.

TARASOVA, Z.N.; EYTINGON, I.I.; SENATORSKAYA, L.G.; FEDOROVA, T.V.;
DOGADKIN, B.A.

Use of phenothiazine (thiodiphenylamine) as an antifatigue agent
for vulcanizates from NK, SKI, and SKS-3QAM. Kauch. i rez.
20 no.9:15-18 S '61. (MIRA 15:2)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti.
(Vulcanization)
(Phenothiazine)

FEL'DSHTEYN, M.S.; CHERNOMORSKAYA, I.G.; EYTINGON, I.I.; GUR'YANOVA, Ye.N.;
DOGADKIN, B.A.

Vulcanizing activity of some 2-mercaptobenzothiazole derivatives
and their exchangeability with radioactive di-2-benzothiazolyl
disulfide. Kauch. i rez. 20 no.10:15-18 0 '61. (MIRA 14:12)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti.
(Vulcanization) (Mercapto group)
(Benzothiazole)

DOGADKIN, B.A.; ZACHESOVA, G.N.; SHOKHIN, I.A.

Reclaiming of rubber by the dispersing method. Kauch. i rez.
29 no.12:15-21 D '61. (MIRA 13:1)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti.
(Rubber, Reclaimed)

S/069/61/023/002/001/008
B101/B208

AUTHORS:

Dogadkin, B. A., Zachesova, G. N., and Shokhin, I. A.

TITLE:

Preparation and properties of aqueous suspensions of vulcanized natural and synthetic rubber

PERIODICAL:

Kolloidnyy zhurnal, v. 23, no. 2, 1961, 150-156

TEXT: The purpose of this study was to investigate the regeneration of used waste rubber by dispersion in aqueous medium. The authors applied the method devised by B. A. Dogadkin and D. M. Pevzner (Ref. 4: Author's certificate no. 29973, 30/IV 1933), in which an oleophilic emulsifier insoluble in water (fatty acid, resinic acid), and then gradually a saponifier (alkali) are added to the hydrocarbon (rubber, plastic, vulcanizate). The reclaimed product is obtained by electrolytic coagulation from the aqueous suspension. The following was studied in the present paper: 1) The effects of the oleic acid, colophony, β -naphthalene sulfonic acid, β -diethersulfonic acid and their sodium salts. The optimum dose was determined by weight. The authors determined the particle size by sedimentation balance in coarse dispersions,

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B101/B208

Preparation and ...

and by means of an electron microscope in fine dispersions (carried out by S. A. Simanovskaya). The following results were obtained:

Emulsifier	g/100 g rubber	particle radius, μ
colophony	10	0.241
oleic acid	10	0.514
β -naphthalene sulfonic acid	5	2.045
β -dinaphthyl-methane-sulfonic acid	5	3.14
dto.	10	4.41

2) Effect of saponifier: NaOH KOH KOH NH₄OH NH₄OH

concentration, % 5 5 7 5 10
average particle radius, μ 0.24 0.22 0.20 is not dispersed

No phase inversion (dispersion of the organic phase in water) occurred in NH₄OH owing to its volatility. The same result was obtained for Na₂B₄O₇, but this is able to replace 2/3 of the alkali, a particle radius of 0.59 μ being obtained. 3) The concentration of the alkali solution exerted the

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B101/B208

Preparation and ...

following effect on dispersion:

concentration, %	2	5	10
time of dispersion	90	50	35 min
amount (g/100 g rubber)	67-83	33	25
required for phase inversion	0.249	0.241	0.555.
particle radius, μ			

4) Large particles were formed when the alkali solution was added too quickly (30 min). Slow addition (90 min) gave a fine emulsion. This is stable if the pH of dispersion is not less than 11.5-12.0. 5) The clearance between the rolls had the following effect:

clearance, mm	0.3	0.5	0.8	1.2
particle radius, μ	0.241	0.539	0.601	0.785

6) The consumption of electric energy during dispersion is compared in Fig. 6 with the amount required to plasticize the mixture. It decreases after adding the alkali solution, and approaches the no-load consumption during phase inversion. 7) Fig. 7 shows the effect of a plasticizing activator, i.e., Renatsite 2, (a preparation containing 42.5% trichloro thiophenol).

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Preparation and ...

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B101/B208

8) the behavior of various types of rubber with highly active carbon black as filler was tested on HK (NK, natural rubber); CKW (SKI, synthetic cis-polyisoprene rubber); CKC-30APM (SKS-30ARM, divinyl styrene rubber), and CK5 (SKB, sodium butadiene rubber). Two groups of samples were used for the purpose: 1) samples prepared according to industrial formulas for tire rubber, 2) samples prepared according to a unified formula so that they differed only in the polymer. Table 3 presents the results. The particle radius was found to depend less on the type of polymer than on the density of the vulcanization network. However, the properties of the reclaimed products obtained by dispersion differ in the individual polymers. The authors will later report on this subject. It is mentioned that the dispersion method described has been used in 1938 at the zavod (plant) "Krasnyy treugol'nik" for the regeneration of used rubber. From 1941 onward, this method has not been applied any longer. Mention is made of F. F. Koshelev and I. A. Tartakovskiy. There are 7 figures, 3 tables, and 7 Soviet-bloc references.

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promyshlennosti
(Scientific Research Institute of the Tire Industry)

Card 4/6

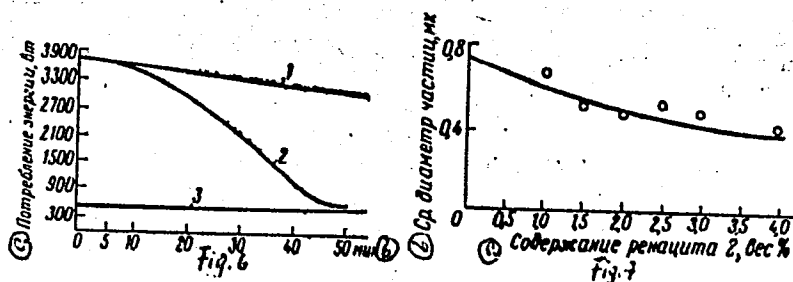
Preparation and ...

S/069/61/023/002/001/008
B101/B208

SUBMITTED: October 26, 1960

Legend to Fig. 6: 1) plasticizing; 2) dispersion; 3) idling; a) power consumption, w; b) min.

Legend to Fig. 7: a) content of Renatsite 2, wt%; b) mean particle diameter, μ .



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B101/R208

① Тип полимера	② НК		③ СКИ		④ СКС-30АРМ		⑤ СКВ	
	1	2	1	2	1	2	1	2
⑥ Группы опытов								
⑦ Число узлов в единице объема вулканизата $\times 10^{-9}$, мл^{-1}	4,03	4,08	3,09	4,88	1,65	4,01	2,39	4,28
⑧ Молекулярный вес участка цепи вулканизата	6000	6000	6850	6450	17 200	7050	11 550	6500
⑨ Средний радиус частиц дисперсии, $\mu\text{м}$	0,428	0,388	0,279	0,362	0,241	0,300	0,270	0,328
⑩ Удельная поверхность частиц дисперсии, $\text{м}^2/\text{г}$	6,16	7,46	8,90	7,45	10,82	8,7	9,80	8,31
⑪ Расход энергии на образование единицы удельной поверхности дисперсной фазы $\times 10^{-4}$, $\frac{\text{квт}\cdot\text{ч}}{\text{м}^2/\text{г}}$	10,9	—	6,5	—	5,44	—	5,90	—

Legend to Table 3: 1) polymer; 2) NK; 3) SKI; 4) SKS-30ARM; 5) SKB; 6) group of experiments; 7) number of lattice points $\times 10^9$ per unit volume of the vulcanizate, ml^{-1} ; 8) molecular weight of the section of the vulcanizate chain; 9) mean radius of disperse particles, μ ; 10) specific surface of disperse particles, m^2/g ; 11) power consumption per unit of specific surface of the disperse phase $\times 10^{-4}$, $\text{kw}\cdot\text{hr}/(\text{m}^2/\text{g})$.

Card 6/6

S/069/61/023/002/008/008
B101/B208

AUTHORS: Dogadkin, B. A., Kargin, V. A., Meyerson, S. I., Rogovin, Z. A.

TITLE: In Memory of Sergey Mikhaylovich Lipatov (Deceased)

PERIODICAL: Kolloidnyy zhurnal, v. 23, no. 2, 1961, 238-239

TEXT: This article is devoted to S. M. Lipatov, an expert in the field of colloid chemistry and physical chemistry of polymers, who died on January 8, 1961. At various institutes he organized laboratories for high-molecular compounds. In particular, he established the laboratoriya iskusstvennogo volokna im. Nauchno-issledovatel'skiy institut im. Karpova (Laboratory of Synthetic Fibers of the Scientific Research Institute imeni Karpov), now the Vsesoyuznyy nauchno-issledovatel'skiy institut iskusstvennogo volokna (All-Union Scientific Research Institute of Synthetic Fibers). In the Soviet Union, Lipatov was the first to lecture on high-molecular compounds and the physical chemistry of dyeing. He was a university teacher for 30 years. Mention is made of his monographs "Fiziko-khimicheskiye osnovy krasheniya" ("Physico-chemical basis of dyeing") (1929); "Vysokomolekulyarnyye Card 1/2

In Memory ...

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soyedineniya" (High-molecular compounds) (1934 and 1943), "Problemy ucheniya o vysokopolimerakh" (Problems of high-polymer research) (1941). Lipatov took part in conferences on colloid chemistry, and was for many years a member of the editorial board of "Kolloidnyy zhurnal" and of the nauchno-tekhnicheskiy sovet Ministerstva pishchevoy promyshlennosti (Scientific and Technical Council of the Ministry of Food Industry). Considerable organizing work was done by Lipatov at the Akademiya nauk BSSR (Academy of Sciences BSSR) as Academician and Vice President. There is 1 figure.

Card 2/2

25887

S/069/61/023/004/002/003

B101/B215

15.9110

AUTHORS:

Pechkovskaya, K. A., Senatorskaya, L. G., Berman, B. Z.,
Dogadkin, B. A.

TITLE:

Reinforcement of rubber in latex. 7. Electron microscopic
examination of filled latex mixtures

PERIODICAL: Kolloidnyy zhurnal, v. 23, no. 4, 1961, 462-463

TEXT: This report was made at the tret'ye Vsesoyuznoye soveshchaniye po elektronnoy mikroskopii (Third All-Union Conference on Electron Microscopy), Leningrad, October 1960. The second communication of this series was published in Trudy II konferentsii po lateksu, Leningrad 1958. The authors based their report on a paper by B. A. Dogadkin et al. (Kolloidn. zh. 18, no. 5, 528, 1956) which shows that a reinforcing action of carbon black in latex can be attained by adding a destabilizing substance (casein) to latex. Here, this effect was studied under an 3M-100 (EM-100) electron microscope having a magnifying power of approximately 20,000. Collodion, quartz, or carbon replicas of the latex film, frozen in liquid nitrogen, were prepared. It was found that 1) all latex films containing neither

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Reinforcement of...

carbon black nor casein had globular structures. 2) Addition of casein changed the structure. Part of the globuli disappeared, and a granular structure formed. Casein removes the protective covering of the globuli, thus allowing the latter to form a continuous polymer phase and to interact with carbon black. The contact area between polymer and carbon black is increased and, thus, causes reinforcement. 3) If the non-vulcanized, filled film was rolled, the last globuli disappeared. 4) Carbon black also had a destabilizing effect upon latex, although to a smaller extent than casein. Films with carbon black without casein contained less but larger globuli. 5) The number of globuli was reduced in the presence of carbon black and casein. [Abstracter's note: The electron microscopic pictures are irreproducible.] There are 1 figure and 2 Soviet-bloc references.

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promyshlennosti, Moskva (Scientific Research Institute of the Tire Industry, Moscow)

SUBMITTED: November 21, 1960

Card 2/2

S/069/61/023/006/002/005
B119/B101

AUTHORS: Dogadkin, B. A., Fel'dshteyn, M. S. Skorodumova, Z. V.

TITLE: Effect of carbon black on the vulcanization kinetics and the character of the sulfur structure of the vulcanizates

PERIODICAL: Kolloidnyy zhurnal, v. 23, no. 6, 1961, 679 - 683

TEXT: Standard type vulcanizates from CKC-30A (SKS-30A) butadiene styrene rubber without filler, and those filled with 50 parts by weight of channel black, furnace black, lamp black, or carbon black of the type fill-black "0", were investigated for their content of bound or replaceable (polysulfide) sulfur. The vulcanizates were also investigated for their capability of swelling. The content of replaceable sulfur was determined by means of radioactive sulfur according Z. N. Tarasova, M. Ya. Kaplunov, M. A. Vas'kovskaya, B. A. Dogadkin (Sb "Vulkanizatsiya rezinovykh izdeliy" (Vulcanization of rubber products), Yaroslavskiy sovnarkhoz, 1960). The effect of the chemical structure of the carbon-black surface on the type of sulfur bond was determined by comparing the effect of untreated channel black (composition: 93.04% C, 1.25% H, 5.71% O, pH 3.47) with that of thermally treated one (at 500°C and Card 1/3

S/069/61/023/006/002/005
B119/B101

Effect of carbon black on...

225 kg/cm² in hydrogen medium; composition: 94.65% C, 1.39% H, 3.96% O, pH 7.2). The possible effect of accelerators (N-cyclohexyl-2-benzothiazole sulfenamide, 2-mercapto benzothiazole, diphenyl guanidine) on the sulfur bond in the presence of the carbon-black types mentioned was investigated with vulcanizates from CKC-3AM (SKS-3AM) butadiene styrene rubber. Results: The content of polysulfide bonds decreases in the order: unfilled vulcanizate (0.6% after 100 min vulcanization), lamp black, fill-black "O", furnace black, channel black (0.2% after 100 min vulcanization). Cross linking is strongest in vulcanizates containing fill-black "O", weakest in those without filler. With decreasing content of oxygen-containing groups on the carbon black surface, the rate of cross linking and the content of bound sulfur increase, while the capability of swelling decreases. The rate of vulcanization and the degree of cross linking (capability of swelling after 100 min vulcanization: without filler: ~400% related to the initial volume of rubber, filled: ~280 - 310%) are higher for vulcanizates with filler than for those without. The effect of fillers is not affected by the accelerator. Vulcanizates with channel black contain least polysulfide sulfur, but are cross-linked in a high degree (low capability of swelling). The

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Effect of carbon black on...

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surface activity of alkaline carbon blacks, especially that of channel black, furthers the formation of free radicals, stronger cross linking taking place through additional C-C bonds between the molecular chains of the rubber. There are 6 figures, 1 table, and 8 references: 6 Soviet and 2 non-Soviet. The two references to English-language publications read as follows: M. L. Studebaker, L. G. Nabors, Rubber. Chem. Techn., 32, 4, 941, 1959; M. L. Studebaker, Rubber, Chem. Techn., 30, 5, 1401, 1957.

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promyshlennosti Moskva (Scientific Research Institute of the Tire Industry Moscow)

SUBMITTED: July 2, 1961

Card 3/3

15.9120

26281
S/074/61/030/008/002/002
B117/B226

AUTHORS: Dogadkin, B. A., and Shershnev, V. A.

TITLE: Vulcanization of rubbers in the presence of organic accelerators

PERIODICAL: Uspekhi khimii, v. 30, no. 8, 1961, 1013 - 1049

TEXT: The present paper was written to complete the survey by D. Craig (Ref. 1: Rubb. Chem. Techn., 30, 1291 (1957)) in which the Soviet, German, and Japanese papers of the last ten years were not considered. When studying the vulcanization the following problems were dealt with: Elementary chemical reactions of vulcanization, mode of action of the accelerators, nature of vulcanization structures and their effect upon the physico-chemical properties of the vulcanization product. For solving these problems both special chemical-analytical procedures and physical methods are used, viz., the optical and electron spectroscopy, isotopic exchange and kinetic studies by radioactive sulfur. Notable results could be obtained in the investigation of the reaction of sulfur with low-molecular model compounds. Two kinds of studies were made: Some of the authors ex-

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Vulcanization of rubbers in ...

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plained the structural changes of rubber during vulcanization mainly by the radical processes. Other scientists consider the elementary reactions as proceeding according to a polar (ionic) mechanism. The different opinions on the vulcanization mechanism do not permit a uniform conception of this complex phenomenon. The reaction mechanism depends on various factors: On the thermodynamic reaction conditions, on the rubber type, and, especially, on the types of accelerator and activator. At present, several vulcanization systems are used: (a) Vulcanization with di- and polysulfides which comprises the following methods: Vulcanization with di-2-benzothiazyl disulfide; vulcanization with sulfur in the presence of di-2-benzothiazyl disulfide; vulcanization with thiuram disulfides; vulcanization with sulfur in the presence of thiuram disulfides and dithio carbamates. (b) Vulcanization in the presence of mercapto-benzothiazole. (c) Vulcanization in the presence of sulfonamides. (d) Vulcanization in the presence of organic bases. Furthermore, papers are discussed which concern the following problems: Effect of binary systems of vulcanization accelerators; structure and activity of vulcanization accelerators; effect of the rubber structure upon its vulcanizability; the part played by vulcanization activators; crosslinking (vulcanization) of rubber solutions at low temperatures; re-

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versibility and the optimum of vulcanization; vulcanization structures and their effect upon the static and dynamic properties and the fatigue of vulcanization products. The following authors are mentioned: S. Ye. Bresler, I. A. Tutorskiy, G. A. Blokh, Ye. N. Gur'yanova, I. Beniska, E. N. Belyayeva, Z. N. Tarasova, A. S. Kuz'minskiy. There are 22 figures, 2 tables, and 112 references: 57 Soviet and 55 non-Soviet. The three most important references to English-language publications read as follows:
Ref. 1: D. Craig, Rubb. Chem. Tehn., 30, 1291 (1957); J. R. Shelton, E. T. McDonel, Lecture at the International Conference on Caoutchouc and Resin, Washington, November 9 - 14, 1959; L. Bateman, R. W. Glasebrook, C. G. Moore, M. Porter, G. W. Ross, R. W. Sawille. Rub. Chem. Techn., 31, 1055 (1958).

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov) X

Card 3/3

GUR'YANOVA, Ye.N.; EYTINGON, I.I.; FEL'DSHEYN, M.S.; CHERNOMORSKAYA, I.G.;
DOGADKIN, B.A.

Structure of some derivatives of 2-mercaptobenzothiazole studied by
the dipole moment method. Zhur. ob. khim. 31 no. 11:3709-3712 N '61.
(MIRA 14:11)

1. Nauchno-issledovatel'skiy institut shinnoy promyshlennosti i
Fiziko-khimicheskiy institut imeni L.Ya. Karpova.
(Benzothiazole---Dipole moments)

S/020/61/138/005/016/025
B103/B215

AUTHORS: Dogadkin, B. A. and Pavlov, N. N.

TITLE: Thermal decomposition of diphenyl guanidine and its interaction with sulfur at vulcanization temperature

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 138, no. 5, 1961, 1111-1114

TEXT: Although diphenyl guanidine (DPG) is one of the most used accelerators of vulcanization guaranteeing high dynamic properties of the vulcanizates, its mechanism so far has not been explained. The authors therefore studied its thermal decomposition at vulcanization temperatures, and its reaction with sulfur. (A) Decomposition of DPG in the melt. Publications reveal that ammonia is liberated by heating DPG at 140°C in argon atmosphere, and that aniline, tetraphenyl melamine, and small amounts of triphenyl dicarbamide are formed. The separation of ammonia is noticeable at vulcanization temperature (136°C) and its rate remains constant up to 145°C. Within the range of 157-180°C, this process is expressed by a reaction equation of the first order. The apparent energy of activation of ammonia formation at these three temperatures is 25.7 kcal

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Thermal decomposition of diphenyl...

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These data show that within 30 min approximately 50 % of the used DPG decompose at 140°C. During the vulcanization of natural rubber (Wistinghausen, Ref. 2: Kautschuk, 5, 57, 75 (1929)), the DPG loss was 40 % after 30 min, and approximately 50 % after 90 min. The zinc oxide in the system considerably affected the rate of DPG consumption. (B) Decomposition of DPG in solution. High-boiling hydrocarbons with different dipole moments were used as solvents: naphthalene 0, xylene 0.62, isopropyl benzene 0.65, glycerin 2.96. The kinetic curves of ammonia formation in melt and solution, at 140°C within 5-6 hr have a similar character. In the initial stage, the individual solvents showed hardly any differences in the rate of ammonia formation. The authors consider this to be an indirect proof of the possible decomposition of DPG following the homolytic mechanism. Furthermore, they assume that no interaction takes place between DPG and the intermediates of its decomposition on the one hand, and polymer molecules on the other. The amounts of ammonia formed by the DPG decomposition in rubber-xylene solution and in the solution of DPG and pure xylene are approximately equal. Measurements of viscosity showed that longer heating of rubber solutions in DPG at 140°C did not change the structure of the polymer. (C) Interaction of DPG with sulfur.

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Thermal decomposition of diphenyl...

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The reaction was conducted in o-xylene at a ratio DPG : sulfur = 1 : 0.25 to 1 : 4. Resinous, nonvolatile products were formed, and H_2S was liberated. The rate of this process is only constant at a molar ratio of DPG : S. The reduction of the S concentration down to 0.25-0.5 moles initiated an induction period. An increase in the S concentration of up to 4 moles causes considerable changes in the kinetic curve of H_2S formation. The authors assume a relation between the two latter phenomena and the formation of $C_{13}H_{13}N_3 \cdot H_2S$ salt complexes if the initial stage of the reaction in the system shows an excess of undecomposed DPG. The equilibrium of formation and decomposition reactions of the complex is shifted towards higher yields of liberated H_2S with increasing sulfur concentration. Two moles of DPG are used for the formation of one mole of H_2S . Nonvolatile reaction products were chromatographically separated on aluminum oxide, and, after purification, individual fractions were spectroscopically examined in the infrared and ultraviolet ranges. The evaluation of infrared spectra is somewhat difficult. Ultraviolet spectra indicate the presence of sulfur-containing groups in the substances

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Thermal decomposition of diphenyl...

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obtained. The curves are similar to those obtained for low-molecular linear sulfides. Clear absorption maxima in the range of 300-380 mμ are probably characteristic of linear polysulfides with 2-5 sulfur atoms. The bands at 1480-1490 cm⁻¹ in infrared spectra, and those at 590-620 mμ in ultraviolet spectra, are assumed to belong to the C=S group. Absorption at 1335-1355 cm⁻¹ is probably due to C₆H₅NH₂. Absorption at 1480-1490 cm⁻¹ and 590-620 mμ corresponds to the thioketo group. The authors therefore assume that an interaction of H₂S with the DPG molecule causing the formation of a thiourea derivative takes place besides the decomposition of DPG and the formation of polysulfides. The formation of the derivative may be represented in a similar way as the interaction of guanidine with water. There are 4 figures and 2 non-Soviet-bloc references.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

PRESENTED: January 28, 1961, by A. A. Balandin, Academician

SUBMITTED: January 26, 1961

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25338

S/020/61/138/006/014/019

B103/B215

15.8060 2209

AUTHORS: Dogadkin, B. A. and Dontsov, A. A.

TITLE: Interaction of polyethylene and sulfur

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 138, no. 6, 1961, 1349-1352

TEXT: The authors studied the interaction of sulfur and high-pressure polyethylene (PE) at 200-250°C. On the basis of their results, this reaction is represented as a radical process in which the eight-membered sulfur cycle is dissociated such: $S_8 \rightleftharpoons \ddot{S}_8 \rightarrow \ddot{S}_x + \ddot{S}_y$ (1). The dissociation of sulfur into radicals is the initial and, simultaneously, the controlling stage. Sulfur biradicals separate hydrogen atoms from methine or methylene groups whereby polymeric and persulfhydryl radicals are formed:

$RCH_2CH_2R_1 + \ddot{S}_x \rightarrow RCH_2\dot{C}HR_1 + HS_x$ (2). The authors assume a subsequent sulfuration and dehydrogenation of the polymer molecules as follows:

$RCH_2\dot{C}HR_1 + \ddot{S}_x \rightarrow RCH_2\dot{C}HR_1$ (3) $RCH_2\dot{C}HR_1 + RCH_2CH_2R_1 \rightarrow RCH_2\dot{C}HR_1 + RCH_2\dot{C}HR_1$ (4).

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Interaction of polyethylene and sulfur

They furthermore assume that polysulfide groups decompose in all stages of the reaction, and that radicals with a reduced number of sulfur atoms are liberated: $\ddot{S}_x \rightarrow \ddot{S}_y + \ddot{S}_z + \ddot{S}$ (5), $\dot{S}_x H \rightarrow \dot{S}_y H + \dot{S}_z H + \dot{S}H$ (6). During the interaction with PE, sulfur is added to PE, i.e., irrespective of the temperature in amounts of 31-37 % of the initial content. In the straight part of the kinetic curve, the reaction rate increases linearly with increasing initial sulfur content. The relative rate shows a reverse dependence. The process is expressed by the equation:

$K = 1.58 \cdot 10^{15} e^{-44.4/RT}$. Hydrogen sulfide (H_2S) is liberated in the reaction. This process is of complicated kinetics. The authors assume that H_2S is

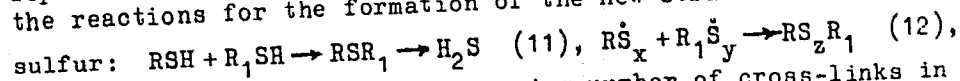
formed by primary reactions between sulfur and PE, and also by secondary reactions in which the added sulfur takes part. The kinetic curves for the H_2S formation showed a sharp bend which forms at a given temperature and an equal amount of added sulfur, irrespective of its content in the initial mixture. H_2S is probably formed by the interaction of sulfhydryl radicals: $2\dot{S}_x H \rightarrow \ddot{S}_y + H_2S$ (7). Its accelerated liberation in the final

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Interaction of polyethylene and sulfur

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stage of the process is due to the reaction of sulfhydryl groups in the PE molecular chains. Besides this intermolecular reaction, intramolecular reactions without the participation of free sulfur may take place, as they are characteristic of the final stage of the process. The linear dependence of the number of cross-links on the sulfur content shows that the reactions for the formation of the new structures are mainly due to



$\text{R}\dot{\text{S}}_x + \text{R}_1 \rightarrow \text{RS}_x\text{R}_1 \quad (13)$. The increasing number of cross-links in a certain stage causes the formation of an insoluble fraction, namely, a gel. Its largest amount is obtained irrespective of the reaction temperature at low amounts (0.6-0.7 %) of bound sulfur, and remains unchanged during further sulfuration. However, the increase in the number of cross-links in the gel proceeds. There is a linear dependence between the swelling maximum (in boiling toluene) and the amount of bound sulfur. Hence, the authors conclude that the cross-linking of molecular chains is mainly due to sulfur-containing groups. After termination of this process, the concentration of cross-links is $5.9-9.4 \cdot 10^{18}/\text{cm}^3$, which means 50-80 sulfur

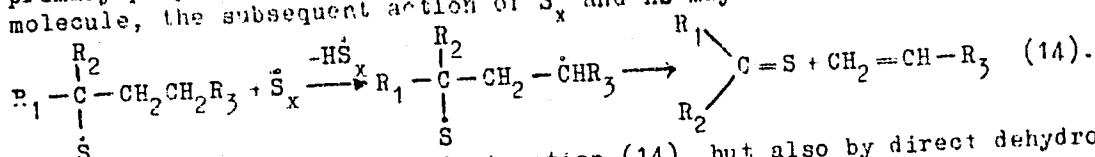
Card 3/6

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Interaction of polyethylene and sulfur

atoms per cross-link. The authors explain this by a kind of destruction and a formation of intramolecular sulfur-containing cycles, and by other kinds of sulfur addition which do not cause cross-linking. If the primary polymer radical forms at the point of ramification of the PE molecule, the subsequent action of \ddot{S}_x and $H\ddot{S}$ may destroy the chain:



Double bonds mainly form by destruction (14), but also by direct dehydrogenation with sulfur: $\ddot{S}_x + RCH_2CH_2R_1 \xrightarrow{\ddot{S}_x} RCHCH_2R_1 + H\ddot{S} \rightarrow RCH=CHR_1 + H_2S + \ddot{S}_{x-1} \quad (15).$

They form at a constant rate. There exists a linear dependence between the amount of added sulfur and the number of resulting double bonds. At the end of the process, their number is only 5-7.5 % of the equivalent amount of liberated H_2S . Theoretically, the amount of H_2S should be equivalent to the sum of resulting cross-links, double bonds, and thion

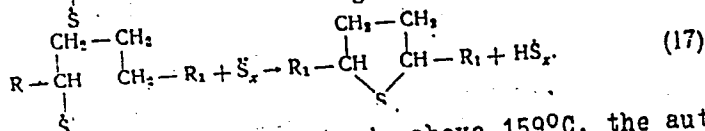
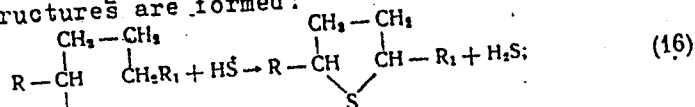
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Interaction of polyethylene and sulfur

groups. H_2S/S , however, is approximately 2, and the sum is much smaller than the equivalent of H_2S . This leads to the assumption that intramolecular ring structures are formed:



Since the polymerization of sulfur sets in above 159°C , the authors assume that some polymer sulfur is contained in sulfurated PE. There are 4 figures and 4 non-Soviet-bloc references. The three references to English-language publications read as follows: W. Friedman (Ref. 2: Refiner and Natural Gasoline Manufacturer, 20, 395 (1941)); F. Faibrother et al. (Ref. 3: J. Polym. Sci., 16, 495 (1955)); D. M. Gardner, G. K. Fraenkel (Ref. 4: J. Am. Chem. Soc., 78, 3279 (1956))

Card 5/6

25338

Interaction of polyethylene and sulfur

S/020/61/138/006/014/019
B103/B215

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov)

PRESENTED: January 28, 1961, by A. A. Balandin, Academician

SUBMITTED: January 26, 1961

Card 6/6

30027
S/020/61/141/001/009/021
B103/B147

15.8620

AUTHORS: Dogadkin, B. A., Tarasova, Z. N., Fogel'son, M. S., and Kashlinskiy, A. I.

TITLE: Interaction of sulfur with rubber under the action of γ - radiation

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 141, no. 1, 1961, 90 - 93

TEXT: The authors studied the interaction of natural-rubber-sulfur (NR + S) mixtures under the action of γ - radiation (dose 6 - 11 Mr) at +20 and -196°C by means of electron paramagnetic resonance (epr). They used a spectrometer with high-frequency modulation at -140 - +20°C. Highly stable radicals were formed by irradiating NR and its mixtures with 2% S; their spectra were equal, their concentration was $(1 - 2.5) \cdot 10^{14} \text{ mg}^{-1}$, and after 30 - 45 days it was still $(0.05 - 0.1) \cdot 10^{14} \text{ mg}^{-1}$. Besides free alkyl radicals formed during irradiation of NR due to the disruption of an H atom and the rupture of

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the -C-C bonds of the NR chains, radicals of the allyl type are also formed. They are stabilized by the effect of conjugation of the free valency with the adjacent double bond, and are assumed to be long-lived polymer radicals. When irradiated at -196°C , the NR spectrum differs from that of the S + NR mixture. Since each spectrum constitutes a superposition of lines, the existence of several radical types is assumed. The inhibitory effect of sulfur may be ascribed, as in benzene, to the delocalization of an electron in the eight-membered ring of the sulfur molecule. When the samples irradiated at -196°C are heated at room temperature for 1 - 1.5 min, their spectrum becomes equal to that of long-lived radicals formed by irradiation of the same samples at $+20^{\circ}\text{C}$. Thus, radicals of varying stability are formed by irradiation at -196°C . The short-lived among them live for a few seconds at room temperature. The concentration dropped by gradual heating of the samples (at intervals of $6-7^{\circ}\text{C}$) from -196 to $+20^{\circ}\text{C}$ in liquid-nitrogen vapor, and keeping the sample at given temperature for 5 min. as well as cooling to -140°C . On

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heating from -196 to -120°C the spectrum was not changed. The range of intense destruction of radicals corresponds to the vitrification range of NR (between -80 and -50°C). The reactivity rapidly increases in the range of the mobility jump of individual links of the molecular chain. Here (as on heating of irradiated NR) only the initial short-lived radicals perish whereas in the S + NR mixture new short-lived radicals with a high g-factor are also formed. This is explained in two ways: (A) At least two new radicals are formed in the mixture, or (B) only one radical with an anisotropic g-factor containing an -S-S group is formed. Since the concentration of newly formed radicals is a function of heating with a maximum at -80°C, it is concluded that at this temperature the ratio of the rate of formation to the rate of destruction of the new radicals is most favorable, effecting a maximum of recordable concentration. For the most distinct additional line characterizing the newly formed radicals, the g-factor is 2.027 ± 0.003 . Its value is equal to the one exhibited by sulfur radicals in the melt at 200°C. It is concluded that the new radicals are due to interaction of S_8 molecules with polymer radicals R^\cdot of

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NR under the action of γ -rays. Below vitrification temperature, this interaction does not take place. It is based on the rupture of the eight-membered sulfur ring, and can only take place at temperatures permitting the required mobility of NR molecular chains: $R^\cdot + S_8 \rightarrow RS_8^\cdot$ (1). RS_8^\cdot

may further decompose with separation of sulfur radicals:
 $RS_8^\cdot \rightarrow RS_{(8-x)}^\cdot + S_x^\cdot$ (2). Thus, S radicals are formed due to interaction of polymer radicals with S molecules at temperatures below 0°C . The radicals $RS_{(8-x)}^\cdot$ live longer than polymeric R^\cdot radicals whereas S_x^\cdot

radicals are more active. The steric structure of rubber is a consequence of the interaction of R^\cdot with each other and with rubber molecules. The structure is formed in a temperature range in which, according to the epr, the radicals disappear most quickly when the irradiated NR thaws. S inhibits the formation of polymer radicals during irradiation. The S-containing radicals can be stabilized by formation of cyclic end groups. Also this process reduces the cross links. An interaction of S_x^\cdot biradicals with molecular chains is possible; nevertheless, intramolecular

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cyclic structures may form which do not increase the number of double bonds. Data of isotopic exchange show that polysulfide linear structures $S_x (x > 1)$ occur in the vulcanizates. These structures increase the static strength of radiation vulcanizates. There are 4 figures and 3 references: 2 Soviet and 1 non-Soviet. The reference to the English-language publication reads as follows: D. Gardner, G. Fraenkel, J. Am. Chem. Soc., 78, 3279 (1956).

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov)

PRESENTED: June 14, 1961, by A. A. Balandin, Academician

SUBMITTED: June 8, 1961

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DOGADKIN, B. A.

USSR

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DOGADKIN, B. A., and TARASOVA, Z. N., Moscow
Institute of Fine Chemical Technology imeni
M. V. Lomonosov /1961 position/- "Influence
of vulcanisation structures on physical and
mechanical properties of vulcanisates"
(Session II)

KUZ'MINSKIY, A. S., LYUECHANSKAYA, L. I.,
FEL'DSHTEYN, L. S., Scientific Research Institute
of Rubber Industry, Moscow /1960 locations/-
"Influence of mechanical stresses on the ageing
of vulcanised rubbers" (Session VIII)
NOVINKO, A. S., GILINSKAYA, N. S., DYUMAYEVA, T. N.,
GRIFACHEVA, A. V., NUKEL'MAN, Z. N., and
GALIL-OGLY, F. A., Scientific Research Institute
of Rubber Industry, Moscow /1961 locations/-
"Investigation of amine vulcanisation of
SKF-26 fluoroco-polymer" (Session II)
REZNIKOVSKIY, M. M., and BRODSKIY, G. I.,
Scientific Research Institute of Tire Industry,
Moscow - "Special features of the mechanism of
abrasion of high-elastic materials" (Session V)

report to be submitted for the 4th Rubber Technology Conference,
London, England, 22-25 May 1962.

S/844/62/000/000/095/129
D204/D307

AUTHORS: Dogadkin, B. A., Tarasova, Z. N., Kaplunov, M. Ya., Kozlov, V. T., Klauzen, I. A. and Matveyev, V. S.

TITLE: The interactions of sulfur with polymers under the action or irradiation

SOURCE: Trudy II Vsesoyuznogo soveshchaniya po radiatsionnoy khimii. Ed. by L. S. Polak. Moscow, Izd-vo AN SSSR, 1962, 554-562

TEXT: The interactions of S with natural rubber, 1,4-cis-polybutadiene, butadiene-styrene and butyl rubber was studied, under irradiation from a Co^{60} source. S added on to natural rubber at 25 - 100°C and to butadiene-styrene rubber and polyethylene at 250°C, under argon, in amounts increasing with the dose (0 - 120 Mr), the rate of addition being faster at higher temperatures. At room temperature the amount of S added on is independent of the initial S-content in the starting mixture (1 - 10% by weight). The addition

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was promoted by C_2Cl_6 . The presence of S hindered the development of structurization, which was, however, promoted by raising the temperature from -80 to 100°C. Pure natural rubber developed cross-linking only up to ~50°C, above which temperature the process was reversed; this reversal was not observed in the presence of 2% S, up to 100°C. The presence of 1 - 4% S in CKC-30AM (SKS-30AM) butadiene-styrene rubber led only to a slight reduction in the degree of cross-linking on irradiation. The loss of unsaturation and $-CH_2-$ groups on irradiation was studied (by ir spectroscopy) on natural rubber both in the presence and absence of S, and was found to be greater in the latter case. The S adds on in a form capable of isotopic exchange with elemental sulfur. Initially 70% of the added sulfur may be exchanged in natural and butadiene-styrene rubbers; this value falls with irradiation to a constant 40% at 50 - 120 Mr. Radiational vulcanizates of natural rubber exhibit increased tensile strength when the polymer contains 2% S, particularly at 100°C; in general, the strength increases with the dose of irradiation. The best strengths were obtained for a mixture of

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natural rubber, S, and C_2Cl_6 . The sulfur is believed to interact with the polymeric radicals⁶ (formed on irradiation by C-C fission) to form polysulfides which (a) lower the thermomechanical stability, and (b) prevent recombination reactions and reactions of radicals formed with C=C, thus hindering the development of branched structures. There are 12 figures.

ASSOCIATION: NII shinnoy promyshlennosti (NII of the Tire Industry)

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DOGADIN, B. A.

S/844/62/000/000/097/129
D234/D307

AUTHORS: Tarasova, Z. N., Dzantiyev, B. G., Yegorov, Ye. V., Kap-
lunov, M. Ya., Petrova, S. B., Sobolev, V. S. and Dogad-
kin, B. A.

TITLE: Investigation of rubber structurization under the action
of accelerated electrons

SOURCE: Trudy II Vsesoyuznogo soveshchaniya po radiatsionnoy khi-
mi. Ed. by L. S. Polak. Moscow, Izd-vo AN SSSR, 1962,
569-575

TEXT: Natural butadiene-styrene and carboxylate rubbers were in-
vestigated. The energy of the electrons was 0.6, 1.6 and 2 Mev. The
specimens were 0.02 - 0.3 mm thick films, 60 x 60 x 1 mm plates
and 10 mm thick washers. Irradiation in free state in air from an
accelerator (0.2 - 0.8 megarad/sec) showed less destruction than
that from a Co^{60} source in inert atmosphere. In natural rubber, des-
truction is much greater in the first case. In filled natural rub-
ber it is less in the first case, in pre-vulcanized mixtures of
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Investigation of rubber ...

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carboxylate rubber it is equal in both cases. Thermomechanical stability of electron-irradiated vulcanized rubbers was about 4 times as high as that of Co^{60} irradiated rubbers. Those of carboxyl containing rubbers show high strength and wear resistance (abrasion index = 115 cm^2/kWh for nonfilled rubbers irradiated with 24 megarad and 200 cm^2/kWh for nonfilled sulphur rubbers). Chemical relaxation curve of these rubbers shows destruction and re-grouping of salt bonds in its initial part. There are 6 figures and 2 tables.

ASSOCIATION: NII shinnoy promyshlennosti (NII of the Tire Industry); Institut khimicheskoy fiziki AN SSSR (Institute of Chemical Physics, AS USSR)

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8/138/62/000/008/005/007
A051/A126

AUTHORS: Berlin, R. L., Dogadkin, B. A., Zachesa, G. N., Korotkova, A. A.,
Linichenko, A. I., Shokhin, I. A.

TITLE: Production of foam rubber articles from latex using aqueous rubber
dispersions

PERIODICAL: Kauchuk i rezina, no. 8, 1962, 14 - 16

TEXT: A method has been developed for the production of foam rubber articles with partial replacement of the latex by aqueous dispersions of old rubber or waste products from foam rubber production. The technique of old rubber dispersion was developed at the НИИШП (NIIShP), whereby the aqueous dispersion of the rubber is a polydispersed colloidal system. Dispersions prepared with colophony as the disperser and 3% aqueous solution of NaOH, as the soaping agent, were used in developing the production method of the latex mix for the foam rubber articles. The latex mix of the foam rubber, based on "revertex-standard" and CKC -50 ПТ (SKS-50PQ) latex, using various types of aqueous rubber dispersions, contained potassium paraffinate, vaseline oil or its emulsion, as the foaming agent, or

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dispersion of vulcanized substances (sulfur, diphenylguanidine, oymate, zinc mercaptobenzothiazol, zinc oxide). The quantity of vulcanizing agents in the mixes was calculated according to the rubber substance of the latex. They also contained a gelatinizing solution (10 - 20% solution of ammonium chloride, 10% solution of ammonia and triethanolamine). The obtained articles met the commercial requirements. The cutting-out process caused no change in the physico-mechanical properties of the foam rubber articles. The latter retain their color when using dispersions produced from foam rubber waste products. It is concluded that by replacing 20 - 30% of the synthetic and natural latex with aqueous dispersions of rubber, the quality of the foam rubber produced by the foaming method, does not drop. According to preliminary calculations, the use of aqueous dispersions of rubber in the production of foam rubber articles should offer considerable technical and economic advantages. There are 2 tables. ✓

ASSOCIATION: Nauchno-issledovatel'skiy institut rezinovykh i lateksnykh izdeliy i Nauchno-issledovatel'skiy institut shinnoy promyshlennosti
(Scientific Research Institute of Rubber and Latex Articles and Scientific Research Institute of the Tire Industry)

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S/190/62/004/001/008/020
B101/B110

AUTHORS: Dogadkin, B. A., Pavlov, N. N.

TITLE: Study of vulcanization in the presence of diphenyl guanidine
II. Thermal decomposition of diphenyl guanidine and its
interaction with sulfur at vulcanization temperature

PERIODICAL: Vysokomolekulyarnyye soyedineniya, v. 4, no. 1, 1962, 52-57

TEXT: The authors studied the formation kinetics of NH_3 during thermal decomposition of the vulcanization accelerator, diphenyl guanidine (DPG), the effect of dipole moments of solvents on the kinetics, and the reaction of DPG with S at 140°C . Tetraphenyl melamine, aniline, triphenyl di-carbamide, and NH_3 , starting at 136°C , were obtained after 5-6 hr decomposition of DPG in the melt in an argon atmosphere. NH_3 forms at a constant rate at 136 and 145°C . Between 157 and 180°C , this process follows a first-order equation. The activation energy is 25.7 kcal/mole. Pure DPG loses about 50% of its weight after 300 min heating at 140°C . Decomposition of DPG dissolved in naphthalene, xylene, isopropyl benzene, or glycerin at 140°C in an argon stream (5-6 hr) showed that the NH_3 formation Card 1/43 ✓

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Study of vulcanization in ...

tion was not affected by the dipole moment of the solvent, but had the same course as in the melt. The amount of NH_3 formed during decomposition of DPG in a xylene solution of rubber was the same as without rubber addition, and the viscosity of the rubber solution remained constant. Hence, DPG and its decomposition products do not react with rubber molecules. DPG and S reacted in o-xylene at various DPG : S ratios and in the presence of stearic acid (SA) (Fig. 3). The induction period observed with a low S content is explained by formation of $\text{C}_{13}\text{H}_{13}\text{N}_3 \cdot \text{H}_2\text{S}$.

The formation of larger amounts of H_2S in the presence of SA is due to the pH change. 2 moles of DPG are used to obtain 1 mole H_2S . Nonvolatile decomposition products were chromatographically separated (Al_2O_3 column), and their IR and UV spectra were taken and interpreted as follows: The absorption band 300-380 $\text{m}\mu$ corresponds to polysulfides $\text{R-S}_x\text{-R}$ ($x = 2-5$); the 1335-1355 cm^{-1} band is probably due to aniline. The 1480-1490 cm^{-1} and 590-620 $\text{m}\mu$ bands correspond to the thioketo group. Hence, DPG is assumed to react with S under formation of diphenyl thiourea. There are 4 figures, 1 table, and 9 references: 3 Soviet and 6 non-Soviet. The two references to English-language publications read as follows: W. I. Hickinbottom, J. Chem. Soc., 1932, 2646; 1934, 1981; 1935, 1279; H.

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Study of vulcanization in ...

S/190/62/004/001/008/020
B101/B110

B. Adams, B. L. Johnson, Industr. and Engng. Chem., 45, 1539, 1953.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov)

SUBMITTED: January 27, 1961

Fig. 3. Effect of the ratio DPG : S and the presence of SA on the formation
of H_2S in o-xylene at $140^\circ C$. (1) 1 : 1; (2) 2 : 1; (3) 1 : 4; (4) 1 : 0.25;
(5) 1 : 1 + 1 mole of SA; (6) 1 : 1 + 3 moles of SA.

Legend: Abscissa: time, hr; ordinate: $C_{H_2S} \cdot 10^2$ g/liter.

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S/138/62/000/005/005/010
A051/A126

AUTHOR: Dogadkin, B.A.; Drozdovskiy, V.F.; Tarasova, Z.N.; Arkhangel'skaya, M.I.

TITLE: Mercaptane and disulfide effect on thermal and thermo-oxidizing destruction of swollen vulcanizates

PERIODICAL: Kauchuk i rezina, ^{vol. 21} no. 5, 1962, 15 - 22

TEXT: The effects of mercaptanes and disulfides on thermal destruction of swollen vulcanizates were studied. The properties of the destruction products were investigated and the substances mainly responsible for the destruction of sulfur bonds of the vulcanizates were determined. It was established that the mercaptanes and the disulfides increase the degree of thermal destruction of the swollen sulfurous vulcanizate, but do not affect the thermal destruction of the sulfurless radiation vulcanizate. Since there is no connection between the destruction rates of the vulcanizate and the oxidation of the solvent in the presence of mercaptanes and disulfides, it is assumed that the rate of the thermo-oxidizing destruction is determined by the effectiveness of the radicals formed.

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Mercaptane and disulfide effect on thermal and

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capable of removing hydrogen atoms from the rubber substance of the vulcanizate. Experimental findings led to the following conclusions: Aromatic and aliphatic mercaptanes and disulfides increase the degree of thermal destruction of the vulcanizate based on SKS-30A rubber. The derivatives of the aromatic row (trichlorothiophenol, β -thionaphthal, disulfide β -thionaphthal and disulfide n-tertiary-butylphenol) are more active than the derivatives of the fatty row (dodecylmercaptane and its sulfide). The mercaptanes are more active than the corresponding disulfides. The trichlorothiophenol, dodecylmercaptane and the disulfide n-tertiary-butylphenol do not noticeably affect the thermal destruction at 180°C of the sulfurless radiation vulcanizate, based on SKS-30A rubber. The rate of the thermo-oxidizing destruction of the vulcanizate depends on the nature of the mercaptanes and the disulfides and that of the solvent. At a constant concentration of oxygen in the system, with a shift of the temperature beyond a certain limit, a reversion of the thermo-oxidizing destruction is noted. The destruction reversion is slowed down in the presence of mercaptanes and disulfides. By comparing the data on the rates of oxidation of the mercaptanes and solvents with that of the thermo-oxidizing destruction of the sulfurous vulcanizate, it is seen that a direct relation between them is not always noted.

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A051/A126

ASSOCIATION: Nauchno-issledovatel'skiy institut shinnoy promyshlennosti (Scientific Research Institute of the Tire Industry)

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